The study of ferroelectric switching using x-ray synchrotron radiation

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Science with Microbeams
APS Scientific Advisory Cross-cut Review
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Outline

- **Introduction**
  - What is a ferroelectric
  - Concentrate on epitaxial films
  - Oxide perovskite system
- **Structural response of epitaxial ferroelectric to electric field**
  - examples of dynamic studies
- **Summary and Conclusions**
  - Domain studies, device studies, future studies require microbeams.

- **Collaborators**

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  - Marian Aanerud (Masters 2002, Physics, NIU)

Materials Science Division
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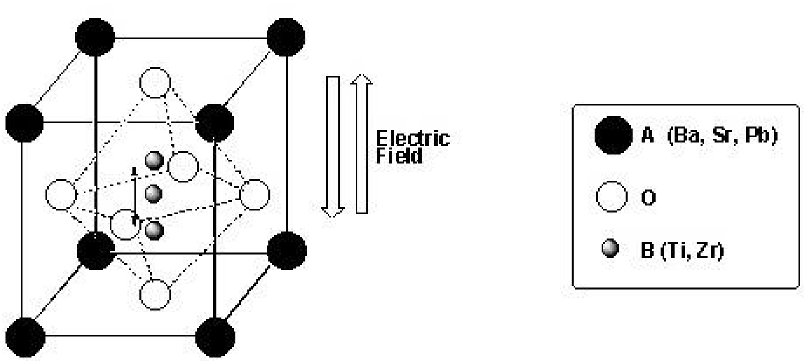
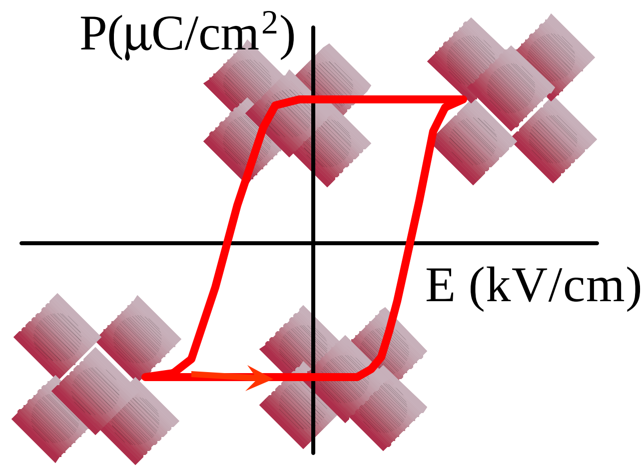
  - W. K. Kee (XFD-XRP,ANL)
  - Armon McPherson, (currently at Sandia)

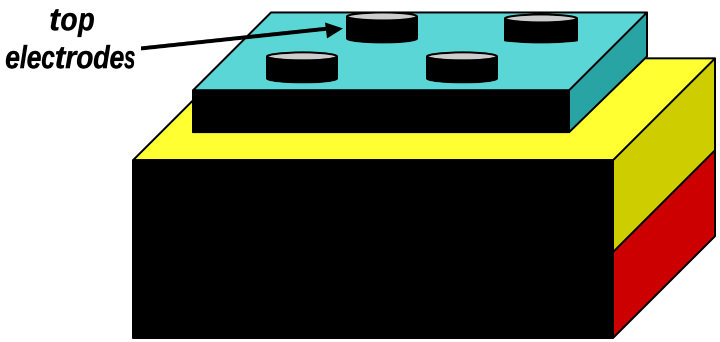
What are ferroelectrics?

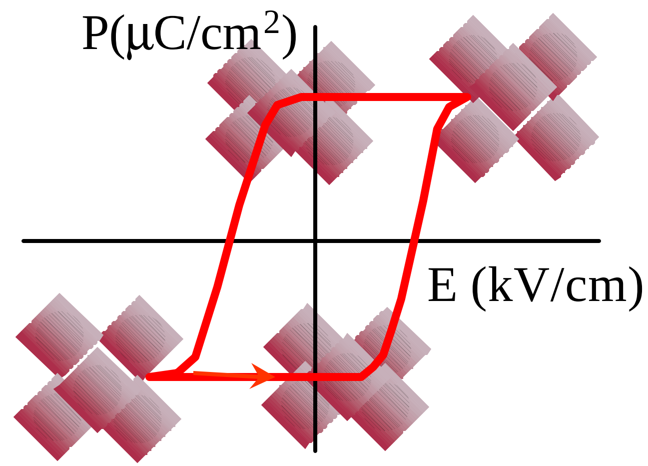
- Spontaneous permanent electric polarization.
- Unit cell of crystal is non-centrosymmetric (charges separated)
- A macroscopic sample with net zero polarization
  - combination of microscopic polarized domains.

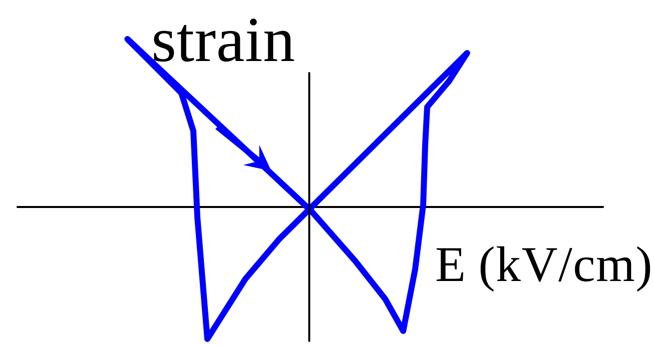




What are ferroelectrics?







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Synchrotron techniques are well matched to the study of the ferroelectric systems

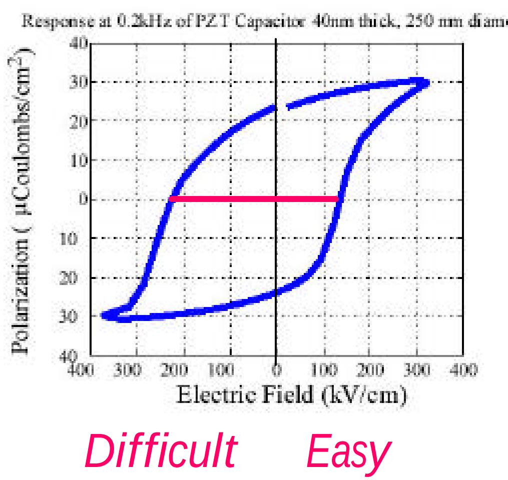
- *Structure-property relationships control:*
  - *dielectric, ferroelectric, piezoelectric, electrostrictive, pyroelectric and electro-optical properties*

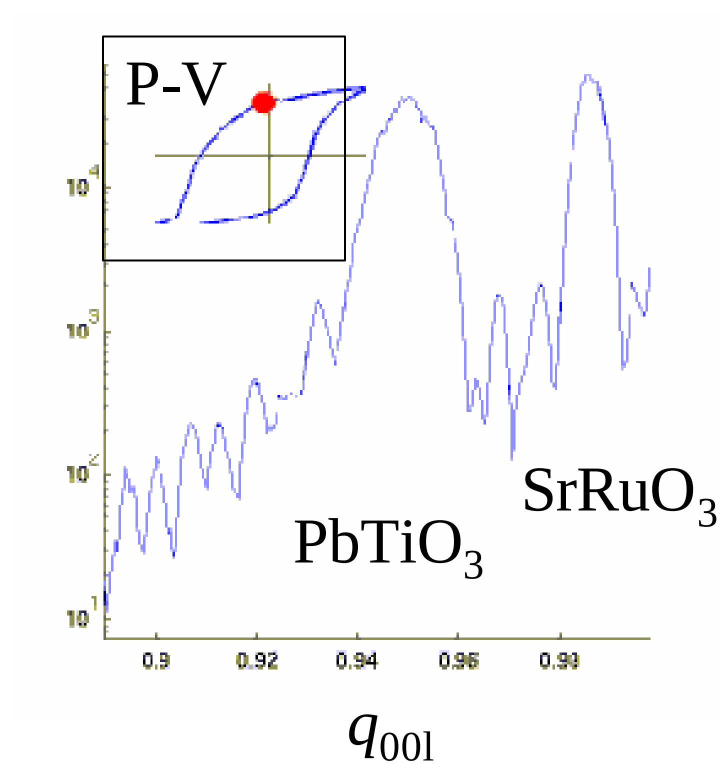
    for actuators, sensors, electro-optical switches, non-volatile memory elements, hi-K dielectric, detectors…

- *Scattering and diffraction examine the structural aspects that control the properties*
  - *Symmetry changes, orientation, lattice parameters, domains configurations*

Scattering example: fingerprints domain evolution

- *Time-resolved scattering*
  - *40 nm $Pb(Ti,Zr)O_3$ film*
  - *200 Hz*





- *Scattering profile can fingerprint the domain configuration in epitaxial films*

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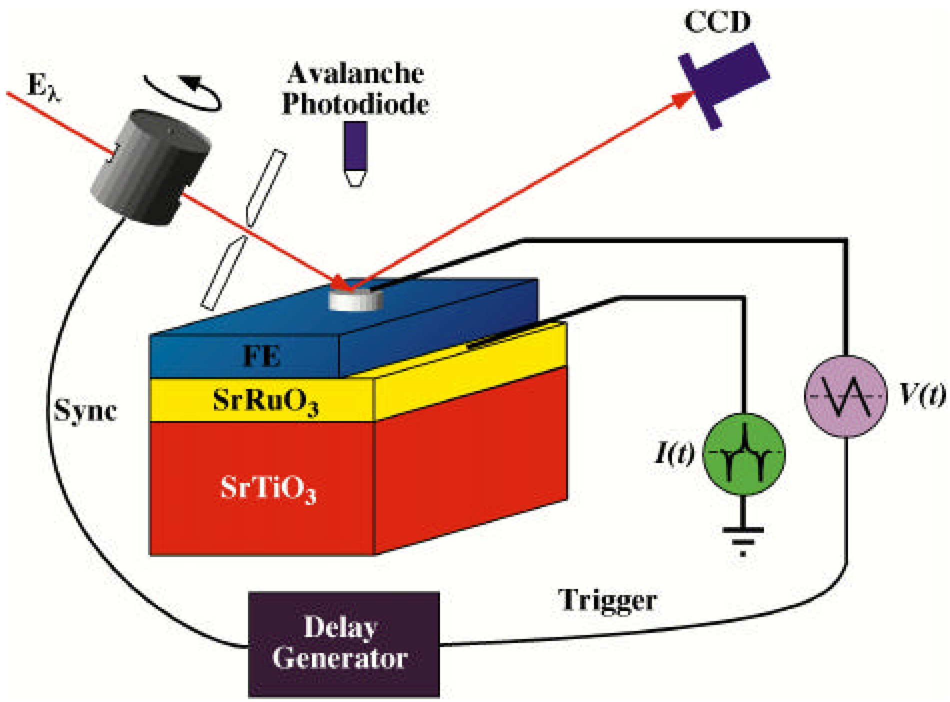
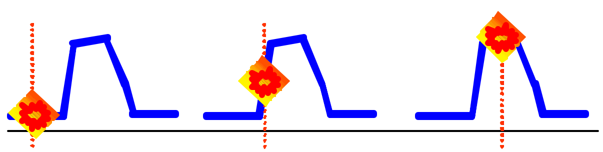
High speed time-resolved Methods (BESSRC 12-ID-D)

- *At each voltage, collect all scattering (area detector)*
- *Utilizes rocking curve of sample to "scan" q*
  - *Chopper synchronized (Hybrid fill: Singlet produces <100 psec x-ray probe pulses*
  - *Electrical stimulation of device synchronized/delayed so that sample is in particular electrical state during exposure*





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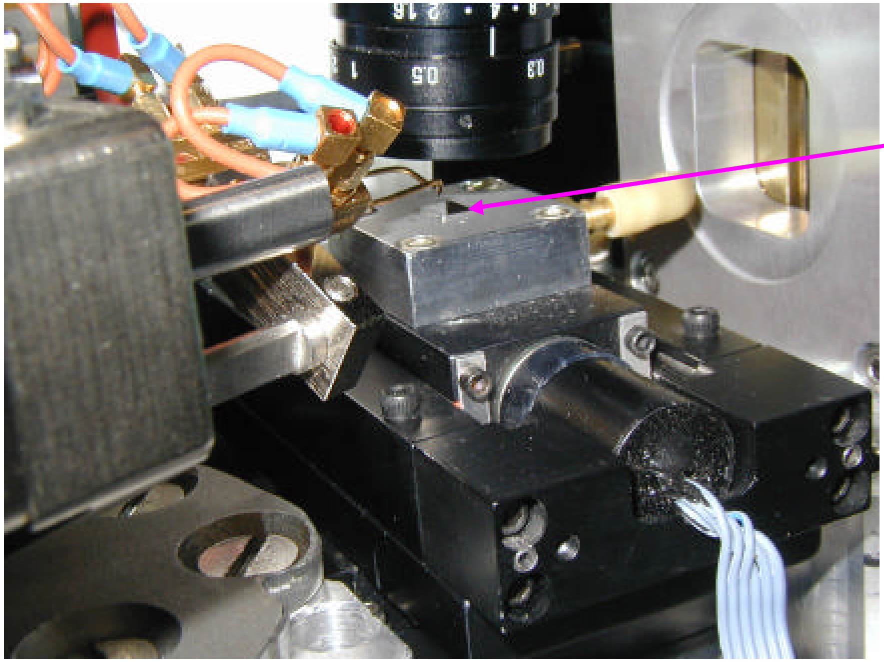
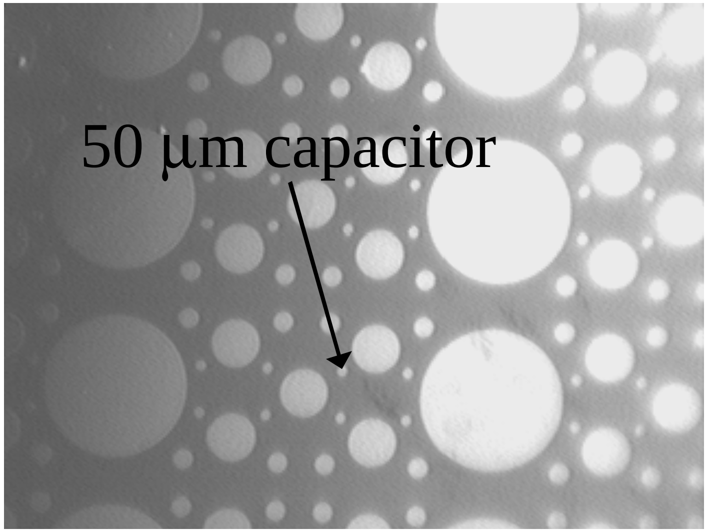
Close-up photograph of sample manipulation and contact region

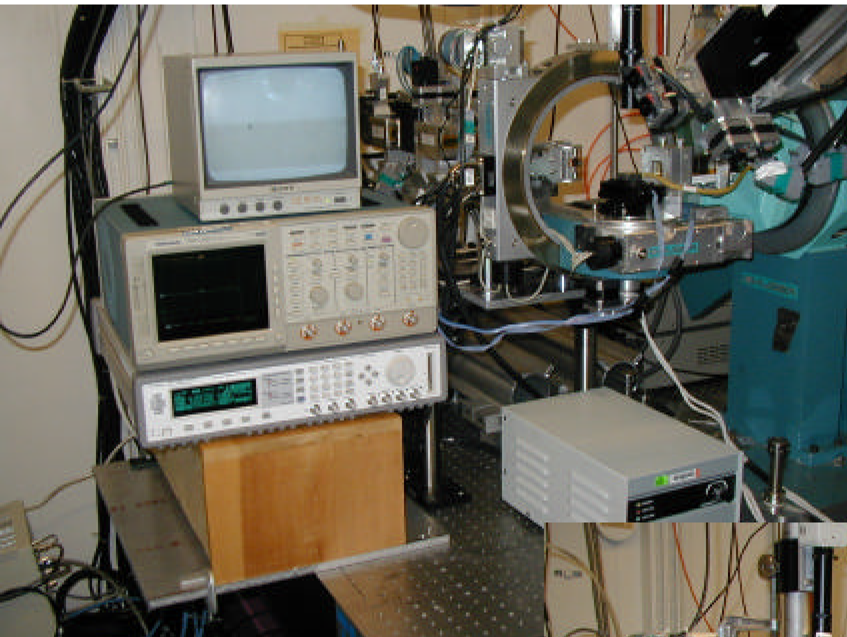


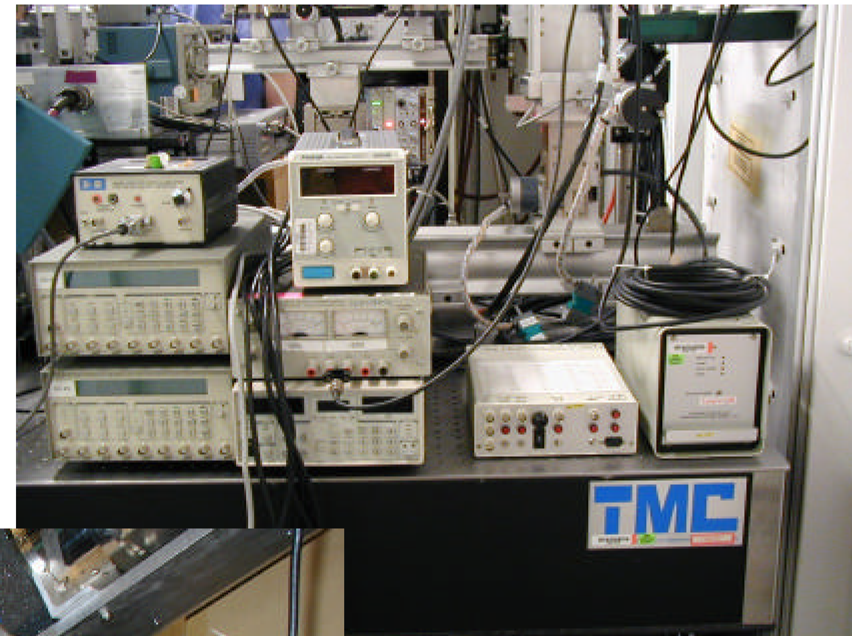


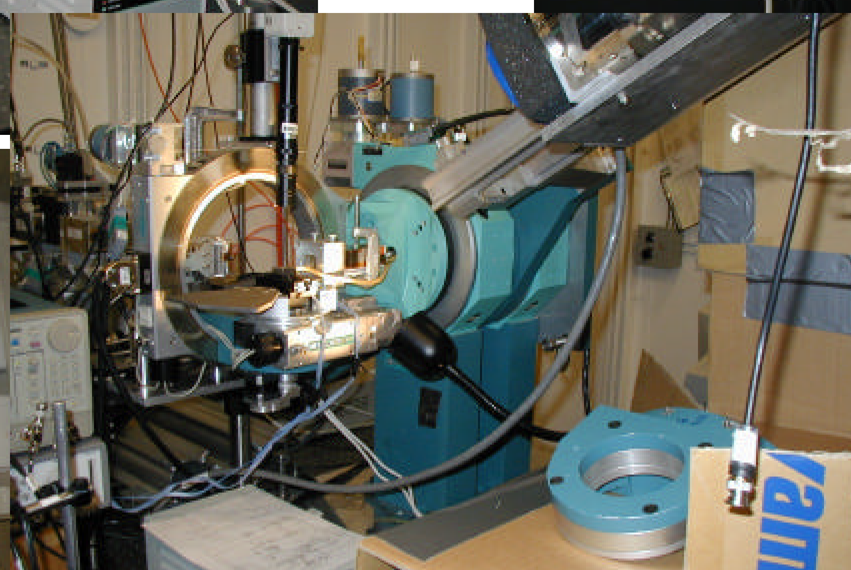
Spot size used: 5µm x 5µm
K-B mirror focus

- X-ray spot must be smaller than the device.
- And x-ray spot must be aligned with the device under electrical stimulation.

More Pictures

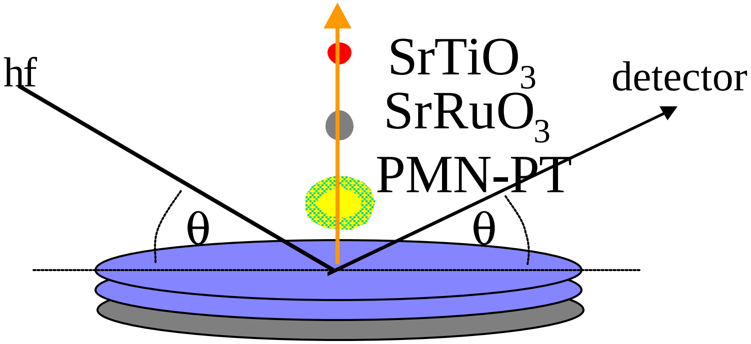




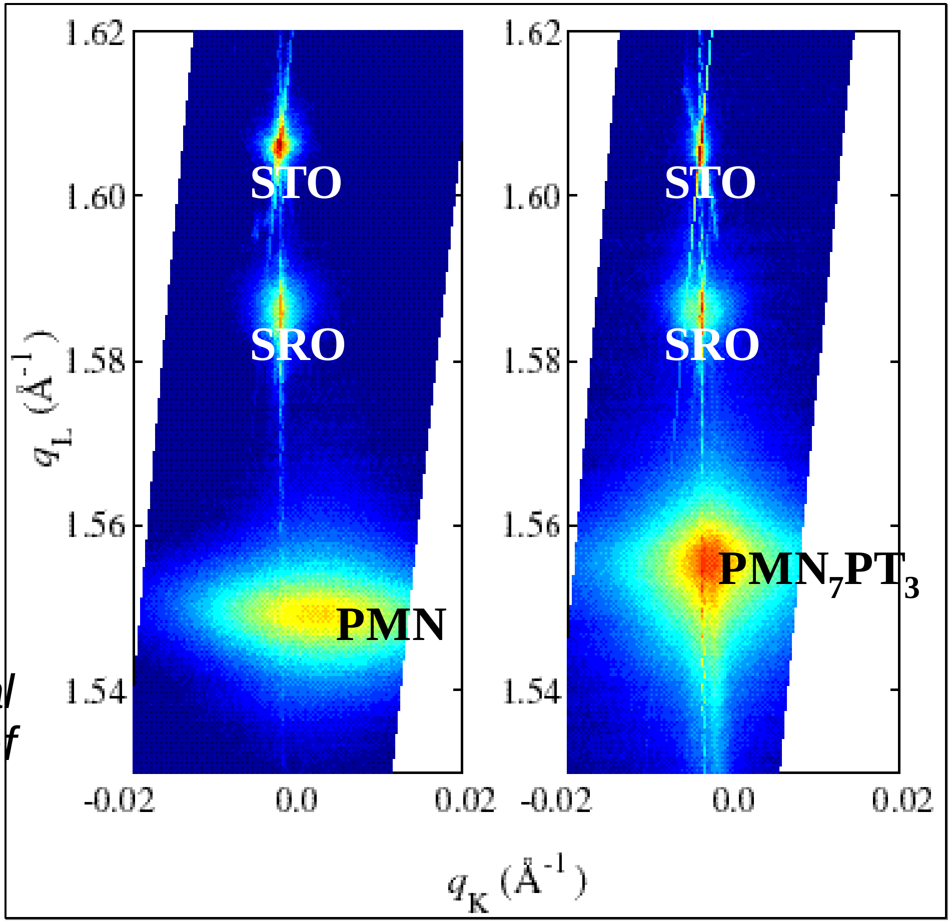


Reciprocal Space Map 001

- *Initial experiments: Focus on position of film Bragg peak region and its immediate neighborhood.*



- *Scattering shown for epitaxial films (thickness ~ 250nm) of PMN and PMN-PT*



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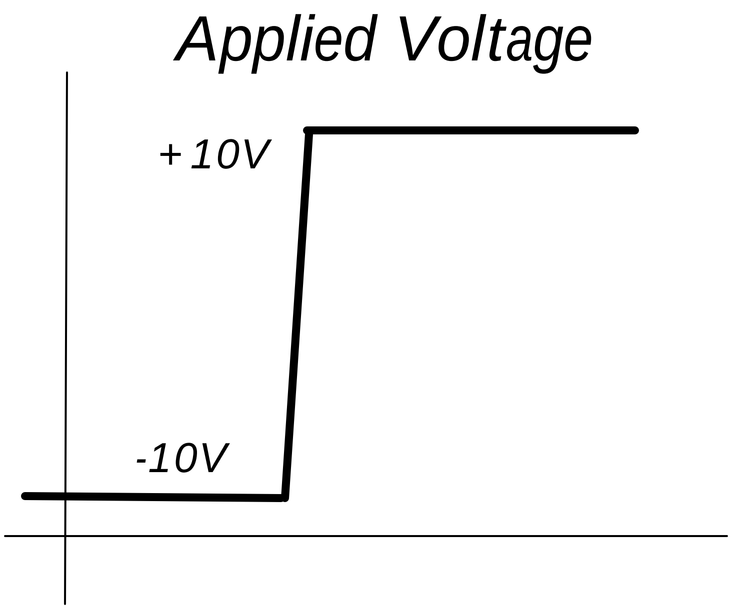
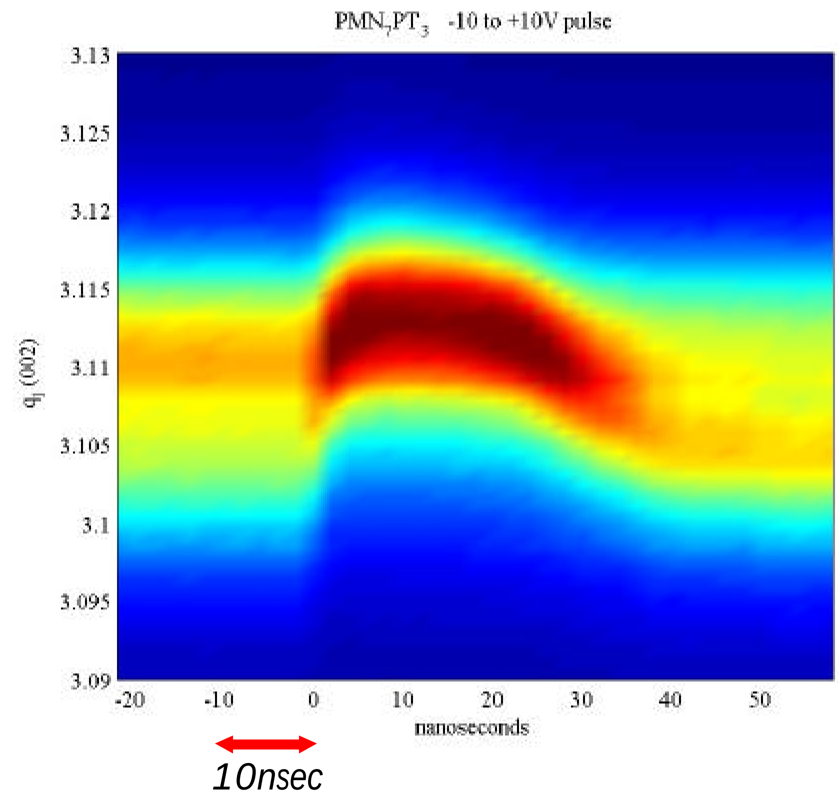
PMN$_7$-PT$_3$ Structural Response to a Step Voltage





- Response (speed) limited by size of device, not by how fast we can measure with x-rays yet
- Smaller devices – smaller beams

Summary and Conclusion

- *Structural techniques available at synchrotrons well suited to ferroelectric systems*
  - *And it's a growing field: see also other groups doing exciting studies of ferroelectric films and crystals using microdiffraction, x-ray topography, and reciprocal space mapping.*

- *Examples from our work:*
  - *Progress in development of techniques to study structural response at 100 psec time scale*
  - *Need to go to smaller devices, embedded devices*

    Progress in switching studies: to 50 μm 'play' device: switching speed limited to ~10nsec

    Smaller devices allow faster switching

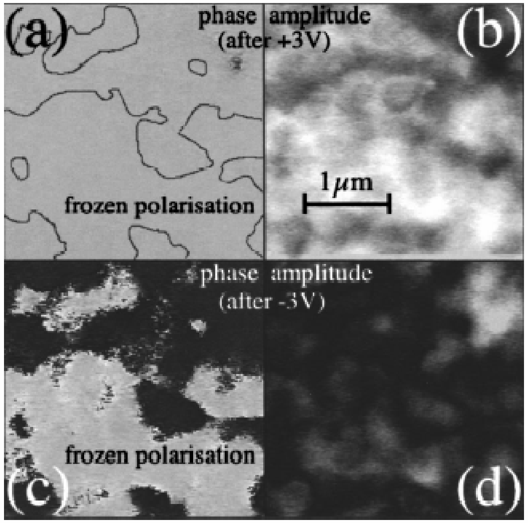
    Need for microbeam capabilities

Preferred Domain Pinning

- Piezo-response atomic force microscopy:
  - Recent direct observation of preferred domain pinning in fatigued ferroelectric films is reported using piezo-response atomic force microscopy.

Direct observation of inversely polarized frozen nanodomains in fatigued, ferroelectric memory capacitors, E. L. Colla, I. Stolichnov, P. E. Bradely, and N. Setter, **Appl Phys. Lett. 82**, 1604 (2003).

Samples: Pt-PZT-Pt films.



After apply

+ 3V

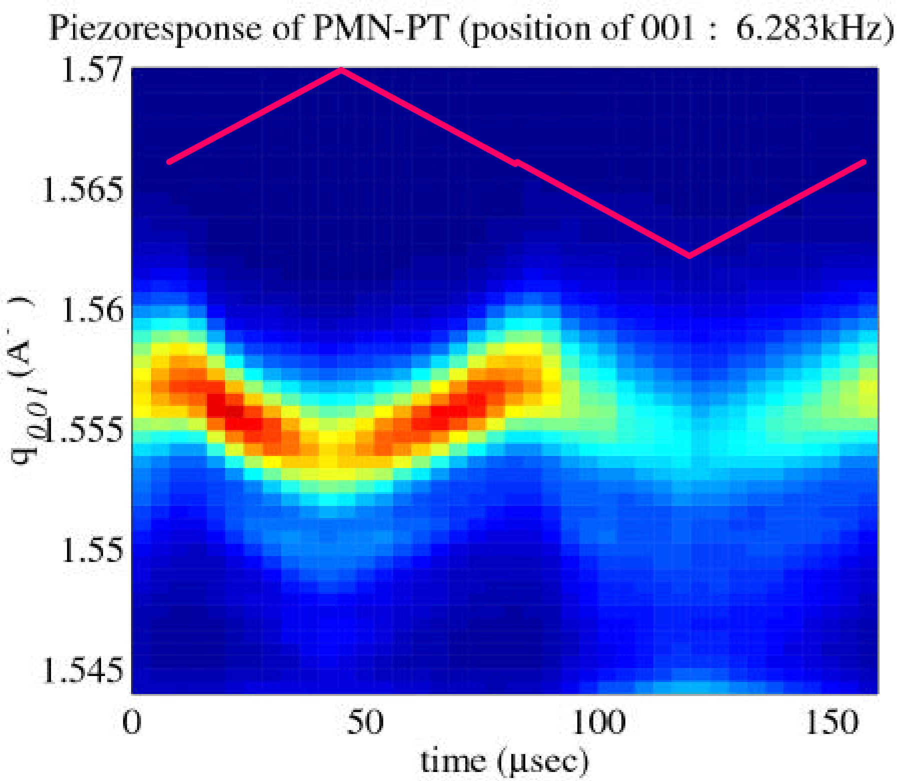
-3V

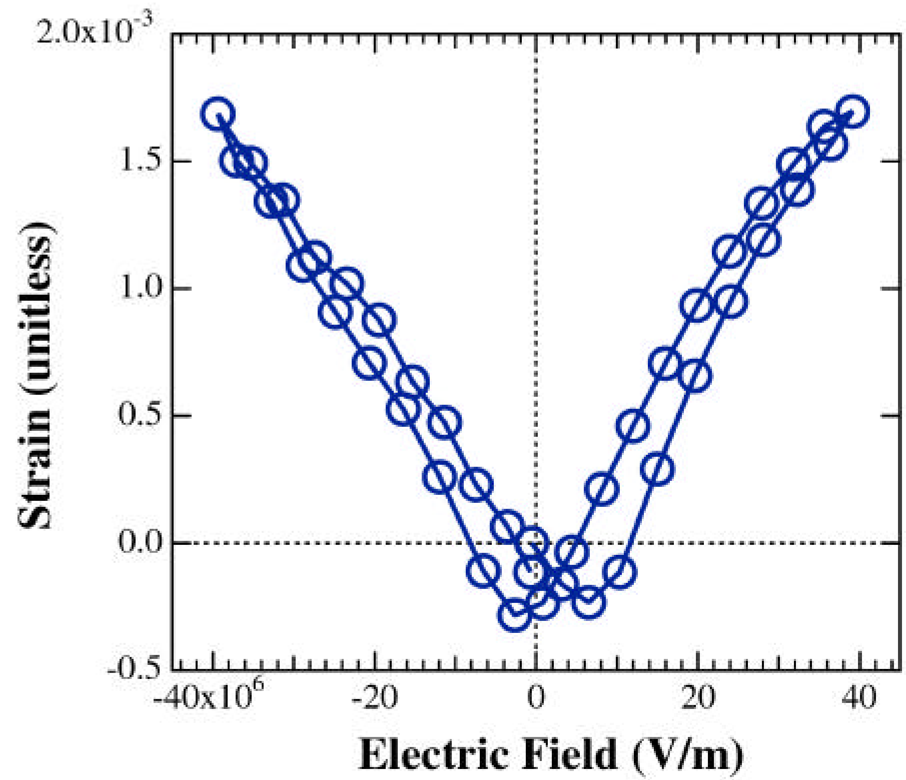
phase amplitude

AFM- Piezoreponse image

Time-Resolved Synchrotron X-Ray Scattering

- Data taken on 250 nm thick PMN-PT film (PT ~ 30-35%)





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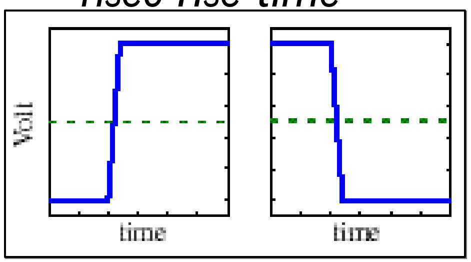
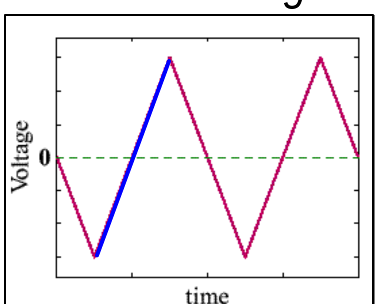
Lattice response: time-resolved x-ray diffraction

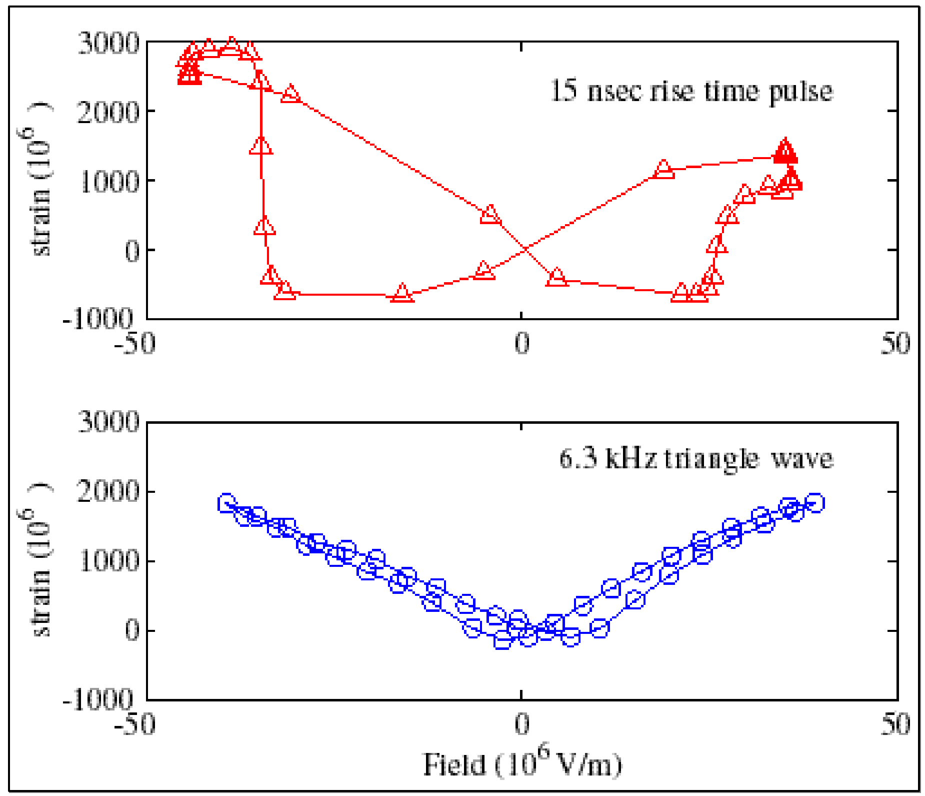
- *Lattice response on different time scales*
  - *pulse with ~15 nsec rise time*



  - *6.3 kHz triangle*



PMN_7-PT_3



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